Pilot-Scale Anaerobic Thermophilic Digester Treating Municipal Sludge

M. A. de la Rubia, L. I. Romero, D. Sales, and M. Perez
Dept. of Chemical Engineering, Food Technology and Environmental Technologies, Faculty of Sea Sciences and Environmental Sciences, University of Cádiz, Campus Río San Pedro s/n, 11510-Puerto Real, Cádiz, Spain

DOI 10.1002/aic.10584
Published online October 24, 2005 in Wiley InterScience (www.interscience.wiley.com).

The work reported concerns anaerobic thermophilic municipal sludge digestion (55°C) in a completely mixed anaerobic digester on a pilot-plant scale (850 L). The experimental protocol was defined to examine the effect of increasing the organic loading rate (OLR) on the efficiency of this digester and to report on its steady-state performance. The reactor had previously been converted from mesophilic to thermophilic conditions following the protocol previously proposed by the authors: this was achieved by a modified method that combined systems investigated in Chicago and in Vancouver. The reactor was subsequently subjected to a program of steady-state operation over a range of sludge retention times (SRTs) of 27, 20, and 15 days. The digester was fed with raw sludge [containing approximately 34.8 g/L volatile solids (VS)] three times per day. Under thermophilic conditions and with a 27-day SRT, the reactor was operated with an OLR of 1.48 kg VS m^{-3} day^{-1}. The solids removal efficiency of the reactor was 42.9%, whereas the volumetric methane production rate in the digester reached 0.35 m^3 m^{-3} day^{-1}. Over an operating period of 150 days, an OLR of 2.63 kg VS m^{-3} day^{-1} was achieved with 41.8% VS removal efficiency in the pilot sludge digester (SRT: 15 days). During this period the volumetric methane production rate in the digester reached 0.20 m^3 m^{-3} day^{-1} and 0.20 m^3/kg VS_r (VS reduction). The greatest efficiency in terms of substrate removal was 54.3% for an OLR of 1.71 kg VS m^{-3} day^{-1} and SRT of 20 days. Under these conditions, the generation of biogas and methane was at levels of 0.86 and 0.58 m^3 m^{-3} day^{-1}, respectively, with a methane yield of 0.70 m^3/kg VS_r. © 2005 American Institute of Chemical Engineers *AIChE J,* 52: 402–407, 2006
Keywords: anaerobic digestion, municipal sludge, solids retention time, thermophilic, volatile fatty acids

Introduction

Anaerobic digestion has been, and continues to be, one of the most widely used processes for the stabilization of wastewater treatment plant sludge. The widespread use of this technique—over and above other stabilization processes—stems from its potential advantages.[1-3] These advantages include the production of energy as methane (in excess of that required for process operation), a reduction of 30 to 50% of sludge volume requiring ultimate disposal, the production of sludge that is generally free from objectionable odors when fully digested, and a high rate of pathogen destruction—particularly in the thermophilic process. However, conventional anaerobic sludge digestion is associated with two well-known problems that have limited its application: digester foaming and low efficiency with respect to volatile solids reduction.[4] Several new processes have been reported for upgrading sludge digestion using thermophilic anaerobic digestion (55°C) and these rep-

Correspondence concerning this article should be addressed to M. A. de la Rubia at mariangeles.delarubia@uca.es.

resent an important alternative to mesophilic anaerobic digestion (35°C).[5-7] In general, thermophilic anaerobic plants offer attractive advantages such as higher solids destruction efficiency, higher biogas production rate, less foaming, and better dewaterability.[2,3,8,9]

Volatile solids (VS) reduction is commonly used to measure the performance of anaerobic digestion processes, and VS content is used as an indicator of the amount of organic matter contained in sludge. Thus, the amount of VS destruction achieved in a sludge stabilization process can be used to measure its effectiveness in stabilizing the organic component of the sludge. The amount of VS reduction achieved depends on the type of sludge digested (primary, waste-activated, trickling filter, or a mixture of these sludges), temperature, and solids retention time (SRT). It is well known that the SRT of a digester is one of the most important factors for the control of anaerobic digestion systems. Although a tremendous amount of research has been performed on the effect of SRT on anaerobic systems, insufficient information is available to clarify the effect of SRT on thermophilic reactor performance.[5,10-12]

On the other hand, the start-up of the process requires a thermophilic inoculum with a balanced growth of various populations of microorganisms. This inoculum is usually obtained from another existing thermophilic plant or, if this is not possible, by adapting a mesophilic system to higher temperatures. The dramatic temperature change involved in this conversion probably selects those subpopulations of bacteria that are at low concentrations in the mesophilic system.[10] Thus, the transition from mesophilic to thermophilic temperatures may require a long acclimation period and may even fail because of the acidic pH. A high sensitivity to temperature changes was found by van Lier et al.[13] when the sludge was adapted over a relatively short period to high temperatures. The stability of the methanogenic stage and the increase in the thermophilic bacteria concentration are much higher if the temperature increases are made very slowly.[2,14]

In the work described here, the strategy proposed by the author[1] for conversion from mesophilic to thermophilic temperatures using a 150-L pilot-scale digester was verified. This protocol involves an initial slow increase from 33 to 43°C at 0.07°C day^{-1}, followed by an abrupt change to 50°C, a period to allow the digester to stabilize, and then a final increase up to 55°C—the temperature at which the system is maintained.

The aims of this study were to investigate the influence of SRT on the performance and treatment efficiency [based on chemical oxygen demand (COD) and VS removal] of sludge digestion in a pilot digester that decomposes municipal sludge under thermophilic conditions (55°C). Furthermore, the protocol selected for conversion from mesophilic to thermophilic operation in the range 35–55°C is described.

Experimental

Description of pilot digester

The pilot-plant scale continuously stirred-tank reactor (CSTR) used in this study had an operational volume of 850 L (Figure 1) and was made from polyester fiber. The temperature was maintained in the range 35–55°C by a tubular heat exchanger. The reactor was inoculated with mesophilic sludge from the wastewater treatment plant (WWTP) located in Jerez de la Frontera, Cádiz, Spain. The main characteristics of the inoculum used are presented in Table 1.

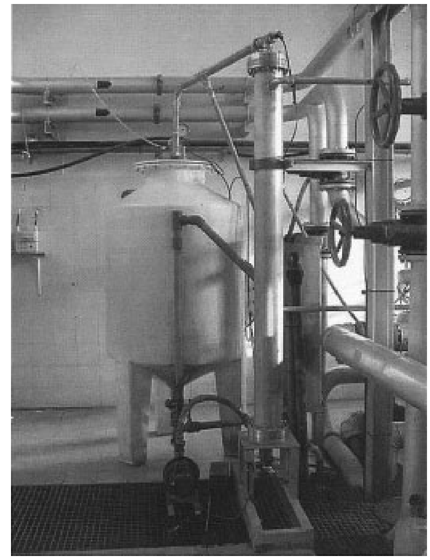

Figure 1. Pilot-plant digester used in this research.

The reactor was fed with prethickened combined primary and secondary waste sludge from the aforementioned WWTP. The feed composition is typical in our location and it remains nearly constant over the 150-day period in which measurement were made and runs carried out.

Initially, in the first operational period under mesophilic conditions, the organic loading rate was 0.91 kg VS m^{-3} day^{-1} with a 27-day SRT. A certain volume of digested sludge (depending on the SRT imposed) was withdrawn from the reactor three times per day, and an equal volume of raw sludge was pumped into the recycle line of the digester through a variable-speed centrifugal pump to provide pseudo-completely mixed conditions in the liquid phase.

In the second operational period, SRT was decreased in steps but was kept constant after each step until steady-state conditions were reached. Attainment of the steady state was verified after an initial period by checking whether the constant effluent characteristic values (VS removal, COD removal, and methane generation) were the mean values of the last measurements in each stage. For the operation at SRT of 27 days, the digester was operated for three SRTs before reaching the steady state.

Finally, in the third operational period, the digester was operated at SRTs of 20 and 15 days and maintained for three SRTs before reaching the steady state.

Sampling and analysis

Parameters measured were as follows: chemical oxygen demand (COD), total solids (TS), and volatile solids (VS) of influent and effluent, pH, individual and total volatile fatty acid (VFA) concentrations, bicarbonate alkalinity of effluent, and gas production and composition of biogas (methane and carbon dioxide percentages).

All analytical determinations were performed according to standard methods.[15]

The volume of gas produced in the reactor was directly

Table 1. Main Characteristics of the Mesophilic Inoculum Used

Parameter	Value
T, °C	36.8
Total solid concentration	47.2 kg/m^3
Volatile solid concentration	36.4 kg/m^3
Total acidity	150 g acetic acid/m^3
Bicarbonate alkalinity	12000 g $CaCO_3$/m^3
pH	7.4

measured by a mass flow sensor, whereas gas composition (methane and carbon dioxide) was determined by gas chromatography according to method previously described by authors.

Concentrations of individual VFA levels in the effluent were determined according to method previously described by authors.

Results and Discussion

First operational period: mesophilic start-up process

For the first 20 days, the digester was maintained at mesophilic temperature and 27-day SRT until a performance level was reached that was analogous to that of the industrial digester in the WWTP Guadalete.

The temperature increase was subsequently performed. The performance and operation parameters for the evolution of the process during the transition from mesophilic to thermophilic temperature are shown in Table 2. This table shows, for each parameter, the final value as the average of the last three data points in each stage of the several operational periods.

Second operational period: conversion from mesophilic to thermophilic process

The strategy initially selected to convert from mesophilic to thermophilic digestion was the protocol proposed by the authors operating with a 150-L pilot-scale digester.[1] This strategy is based on a slow and gradual temperature increase up to 43°C. The procedure attempted by Garber et al.[8] to obtain a thermophilic culture involved raising the temperature of the digester at a rate of 0.6°C day^{-1} until the desired temperature was reached. This method, however, "soured" the digester. In an effort to overcome this difficulty, the temperature of the digester in this study was increased at 0.07°C day^{-1}. Furthermore, the digester was operated at variable retention time during the temperature adjustment. A temperature of 45°C was avoided and the system was adjusted to 50°C very rapidly.

In this way, after mesophilic conditions had been reached, the temperature of the digester was increased in 113 days from 35 to 43°C. The SRT was maintained at 27 days. At this time the mesophilic microorganisms were active. Thus, the organic removal rate was 0.56 kg VS m^{-3} day^{-1} (1.09 kg COD m^{-3} day^{-1}) and the volatile solids destruction efficiency and organic removal efficiency were 42.0 and 48.3%, respectively. The biogas production rate increased until it reached 0.54 m^3 m^{-3} day^{-1}. At this stage, the VFA concentration increased from 261 to 281 mg acetic acid/L at the end of this period. The volatile acidity/alkalinity ratio was 0.014 and the pH was maintained at 7.4.

The temperature of the digester was subsequently increased rapidly to 50°C to allow the digester to stabilize. Incremental temperature increases were not used because a temperature of 45°C was found to be unstable.[1] During the following 31 days, the temperature was maintained at 50–52°C and the SRT imposed was 48 days with an organic loading rate of 0.83 kg VS m^{-3} day^{-1} (1.41 kg COD m^{-3} day^{-1}).

The organic removal rate was 0.41 kg VS m^{-3} day^{-1} (0.67 kg COD m^{-3} day^{-1}) and the volatile solids destruction efficiency and organic removal efficiency were 49.7 and 47.2%, respectively. Both the gas production rate and yield of methane decreased, with the latter having a value of 0.26 m^3/kg VS$_r$ (VS reduction). The VFA content increased sharply to 632 mg acetic acid/L at the end of this period. The volatile acids/alkalinity ratio was 0.056 and the pH increased to 7.5.

The temperature was subsequently maintained at 50°C and the SRT was reduced to a 27-day SRT over 32 days. The organic loading rate increased (1.35 kg VS m^{-3} day^{-1} and 2.60 kg COD m^{-3} day^{-1}) and high levels of removal were maintained (44.5 and 43.1% of volatile solids destruction and organic removal efficiency, respectively).

Biogas and methane generation levels increased to 0.49 and 0.32 m^3 m^{-3} day^{-1}, respectively. The VFA content increased again to 757 mg acetic acid/L. The reactor operated at 50°C and 27-day SRT for 32 days and the temperature was then increased from 50 to 55°C over 2 weeks while the SRT was maintained (27 days). This temperature adjustment was started on the 25th week of the experiment.

When the steady state was achieved (after 81 days) the operation and performance parameters of digestion were as

Table 2. Summary of Performance Parameter Data for the Digester During the Transition and Steady-State Operation, at Each SRT

Operational Period	Stage	T (°C)	SRT (day)	pH	OLR (kg VS m^{-3} day^{-1})	ORR (kg VS m^{-3} day^{-1})	% VS$_r$	Biogas (m^3 m^{-3} day^{-1})	ac/alk*	Total Volatile Fatty Acid (g acetic acid/m^3)
1	Mesophilic, 35°C	35.2	27	7.3	0.91	0.46	50.5	0.35	0.04	261
2	Increasing 35–43°C, 0.07°C/day	—	27	7.4	1.29	0.56	42.0	0.54	0.01	281
	Maintenance at 50°C	50.0	48	7.5	0.83	0.41	50.0	0.16	0.06	632
	Maintenance at 50°C	49.8	27	7.5	1.35	0.60	44.5	0.49	0.06	757
	Thermophilic, 55°C	55.0	27	7.5	1.48	0.64	42.9	0.57	0.09	1791
3	Thermophilic, 55°C	55.1	20	7.5	1.71	0.94	54.3	0.86	0.12	1690
	Thermophilic, 55°C	55.0	15	7.5	2.63	1.11	41.8	0.35	0.23	4448

*Acidity to alkalinity ratio.[15]

Table 3. Individual VFA Levels (Acetate, Propionate, C-4, and Total VFA, as g/m^3) in the Effluent of the Reactor under Mesophilic and Thermophilic Conditions

Stage	SRT	Acetic Acid	Propionic Acid	C4 Acids (butyric + isobutyric)	Total Acidity (g acetic acid/m^3)
Mesophilic 35°C	27	245	13	0	262
Thermophilic 55°C	27	598	593	140	1791
Thermophilic 55°C	20	695	418	124	1690
Thermophilic 55°C	15	1414	1195	433	4448

follows: the OLR was 1.48 kg VS m^{-3} day^{-1} (2.67 kg COD m^{-3} day^{-1}) and the volatile solids destruction efficiency and organic removal efficiency were 42.9 and 39.5%, respectively. The biogas production rate increased to reach 0.57 m^3 m^{-3} day^{-1} (0.35 m^3 CH_4 m^{-3} day^{-1}) with a yield of 0.56 m^3/kgVS$_r$. The VFA content increased to about 1791 g acetic acid/m^3 at the end of this period. The volatile acids/alkalinity ratio reached 0.09 and the pH was 7.5.

The individual VFA levels (acetate, propionate, C-4, and total VFA, as g/m^3) in the effluent of the reactor under mesophilic and thermophilic conditions are shown in Table 3. As can be observed, the acetic acid level increased from 245 to 598 g/m^3 when the temperature was raised from 35 to 55°C. The remarkable increase in propionic acid from 13 to 593 g/m^3 is also worth noting.

Third operational period: performance of digester under thermophilic conditions (20- and 15-day SRTs).

At this stage the experimental protocol was defined to examine the effect that increases in the organic loading rate have on the efficiency of the digester and to report on its steady-state performance at 20- and 15-day SRTs.[1] The attainment of the steady state was verified after an initial period (three times the SRT) by checking whether constant effluent characteristic values were obtained (VS destruction, COD removal, methane generation, and individual VFA levels).

The progress of the digestion process was assessed by monitoring COD and VS reduction (COD$_r$ and VS$_r$), gas production and gas composition, pH, and total and individual VFA levels.

The solids retention time was decreased stepwise from 27 to 15 days. The organic volumetric loading and removal rates, OLR and ORR, were between 1.48 and 2.63 kg VS m^{-3} day^{-1} (2.67 and 1.06 kg COD m^{-3} day^{-1}). VS removal was observed to increase to 54.3% when the SRT was 20 days. The efficiency subsequently decreased to 41.8%. In terms of COD, the removal efficiency was 53.0% at the 20-day SRT and decreased sharply at 15 days, with a value 25.0% COD$_r$ obtained. As expected, the highest overloading caused the most dramatic changes because it is likely that the population size would have to increase under such organic loading rate conditions.

The effect of organic loading rate (expressed as kg VS m^{-3} day^{-1} and kg COD m^{-3} day^{-1}) on volumetric biogas production activity (as m^3 m^{-3} day^{-1}) is represented in Figure 2. On the other hand, methane production averaged 0.35, 0.58, and 0.20 m^3 m^{-3} day^{-1} at SRTs of 27, 20, and 15 days, respectively. The methane yield, expressed as liters of methane produced per gram of VS$_r$, varied from 0.56, to 0.70, to 0.20 m^3 CH_4/kg VS$_r$. Once again, it was found that the pilot-plant performance at the 15-day SRT is associated with a significant decrease in the treatment efficiency because the microbial population does not increase at the same time as the organic loading rate.

The pH was around 7.5 throughout all the stable processes.

Individual and total VFA concentrations were quite high, as can be observed from the results in Table 3. The acetic acid level increased on decreasing the applied SRT and, similarly, the propionic acid level increased until it reached approximately 1200 g/m^3.

The acidity/alkalinity relationship decreased with SRT until stabilization, at which point constant values of around 0.23 g acetic/g calcium-bicarbonate (a very high value for thermophilic conditions) were reached.

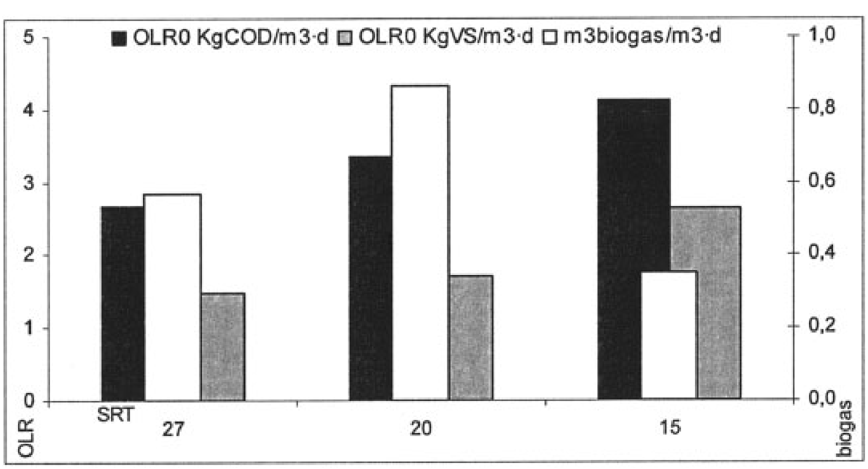

Figure 2. Effect of organic loading rate (expressed as kg VS m^{-3} day^{-1}and as kg COD m^{-3} day^{-1}) on volumetric biogas production rate (as m^3 m^{-3} day^{-1}).

Comparison of mesophilic–thermophilic processes

A thermophilic process can support a high loading rate in comparison with a mesophilic process. Thus, the system can operate at 2.63 kg VS m^{-3} day^{-1} (SRT: 15 days) and maintain 41.8% VS_r. Under mesophilic conditions on the industrial scale, a volatile solids reduction of 40% is an acceptable performance value in the sludge digestion process.[16] In the present study, thermophilic digestion of municipal sludge was established at 4.12 kg COD m^{-3} day^{-1} (15 days) and gave a reduction in volatile solids of 42%.

A higher degradation efficiency is associated with increased gas production and improvements in the energy balance of the process. The greatest efficiency in terms of the methane production rate was 0.58 m^3 m^{-3} day^{-1} for an OLR of 1.71 kg VS m^{-3} day^{-1} (0.70 m^3 CH_4/kg VS_r) at a 20-day SRT.

One of the major drawbacks concerning the use of thermophilic digestion is that the final effluents contain higher concentrations of VFAs than those from a mesophilic digester. The data reported here support this supposition (at 27-day SRT, total acidity was about 1791 g/m^3 under thermophilic conditions vs. 261 g/m^3 under mesophilic conditions). It can be seen from the results in Table 3 that the individual mesophilic volatile acid concentrations were always in the range 0–250 g/m^3. Under thermophilic conditions and 15-day SRT, acetic and propionic acid concentrations were in the range 1200–1500 g/m^3. Therefore, the individual VFA levels were consistently lower than those of the thermophilic unit.

High total VFA values in thermophilic processes have also been reported by Dinsdale et al.[17] They operated continuous thermophilic studies on coffee waste over long periods and achieved stable digestion at a variety of loading rates. However, they did find that some experiments began to show increasing levels of volatile fatty acids after a certain time of operation. However, Kiyohara et al.[18] reported that the thermophilic process has an advantage in treating raw sludge under high loading rates compared with the mesophilic process. The activity of thermophilic bacteria was higher than that of mesophilic bacteria,[19] although thermophilic bacteria tend to remove propionic acid more slowly than mesophilic bacteria.[5,18] Similar results were also observed in the research described here.

Conclusions

Anaerobic thermophilic municipal sludge digestion (55°C) in a completely mixed anaerobic digester on a pilot-plant scale (850 L) is studied. The municipal sludge used has a typical composition in our location and it remains nearly constant over the 150-day period in which measurements were made and runs carried out.

The reactor had previously been converted from mesophilic to thermophilic conditions following the protocol proposed by the authors.[1] The proposed protocol is successful in using a very slow increase in temperature followed by an increase in organic loading and, finally, a decrease in the solid retention time (SRT).

Experimentally it was confirmed that the thermophilic sludge pilot digester can achieve removal levels > 54% VS and 53% COD reduction at loading rates of 1.71 kg VS m^{-3} day^{-1} and 3.34 kg COD m^{-3} day^{-1} under steady-state conditions (SRT: 20 days).

The greatest OLR applied was 2.63 kg VS m^{-3} day^{-1} and this gave rise to a substrate removal efficiency rate > 42% VS.

A higher degradation efficiency is associated with increased gas production and improvements in the energy balance of the process. The greatest efficiency in terms of the methane production rate was 0.58 m^3 m^{-3} $day^{-1}$for an OLR of 1.71 kg VS m^{-3} day^{-1} and a methane yield of 0.70 m^3 CH_4/kg VS_r at a 20-day SRT.

High values of total and individual VFA concentrations in the effluent were found during all periods of operation and the digester showed stable operation.

The thermophilic process can support higher loading rates than the mesophilic process. The activity of thermophilic bacteria was higher than that of mesophilic bacteria.

Acknowledgments

This research was supported by a grant from the Comisión Internacional de Ciencia y Tecnología (CICYT) of the Spanish Government (PETRI, ref. 95-0208-OP, Madrid, Spain).

The authors thank the staff at the Guadalete Wastewater Treatment Plant in Jerez de la Frontera, Cádiz, Spain (Aguas de Jerez Empresa Municipal S.A. and PROSEIN) for their helpful cooperation.

Literature Cited

1. de la Rubia MA, Romero LI, Sales D, Pérez M. Temperature conversion (mesophilic to thermophilic) of municipal sludge digestion. *AIChE Journal.* 2005;51(9):2581–2586.
2. Rimkus R, Ryan J, Cook E. Full scale thermophilic digestion at the west–southwest sewage treatment works, Chicago, Illinois. *J Water Pollut Control Fed.* 1982;54:1447-1457.
3. Peddie CC, Tailford J, Hoffman D. Thermophilic anaerobic sludge digestion: Taking a new look at an old process. Proc of 10th Annual Residuals Biosolids Management Conference of the Water Environment Federation. Vol. 1. 1996:39-46.
4. Li YY, Noike T. Upgrading of anaerobic digestion of waste activated sludge by thermal pre-treatment. *Water Sci Technol.* 1992;26:857-866.
5. Zabranska J, Dohanyos M, Jenicek P, Kutil J. Thermophilic process and enhancement of excess activated sludge degradability—Two ways of intensification of sludge treatment in Prague central wastewater treatment plant. *Water Sci Technol.* 2000;41:265-272.
6. Ahn JH, Forster CF. A comparison of mesophilic and thermophilic anaerobic upflow filters. *Bioresource Technol.* 2000;73:201-205.
7. Pérez M, Romero LI, Sales D. Organic matter degradation kinetics in an anaerobic thermophilic fluidised-bed bioreactor. *Anaerobe.* 2001; 7:25-35.
8. Garber WF, Ohara GT, Colbaugh JE, Raksit SK. Thermophilic digestion at the Hyperion Treatment Plant. *J Water Pollut Control Fed.* 1975;47:950-961.
9. Pérez M, Romero LI, Sales D. Comparative performance of high rate anaerobic thermophilic technologies treating industrial wastewater. *Water Res.* 1998;32:559-564.
10. Zhang TC, Noike T. Influence of retention time on reactor performance and bacterial trophic populations in anaerobic digestion processes. *Water Res.* 1994;28:27-36.
11. Maharaja I, Elefsiniotis P. The role of HRT and low temperature on the acid-phase anaerobic digestion of municipal and industrial wastewaters. *Bioresource Technol.* 2000;76:191-197.
12. Miron Y, Zeeman G, Van Lier JB, Lettinga G. The role of sludge retention time in the hydrolysis and acidification of lipids, carbohydrates and proteins during digestion of primary sludge in CSRT systems. *Water Res.* 2000;34:1705-1713.
13. Van Lier JB, Hulsbeek J, Stams AJ, Lettinga G. Temperature susceptibility of thermophilic methanogenic sludge: Implications for reactor start-up and operation. *Bioresource Technol.* 1993;43:227-235.

14. Garber WF. Operating experience with thermophilic anaerobic digestion. *J Water Pollut Control Fed.* 1982;54:1170-1175.
15. APHA (American Public Health Association, American Water Works Association, Water Pollution Control Federation). In: Clesceri LS, Greenberg AE, Trussell RR, eds. *Standard Methods for the Examination of Water and Wastewater*. 17th Edition. Washington, DC: APHA; 1989.
16. de la Rubia MA, Pérez M, Romero LI, Sales D. Effect of solids retention time (SRT) on pilot scale anaerobic thermophilic sludge digestion. *Process Biochemistry.* 2005 (in press, available online).
17. Dinsdale RM, Hawkes FR, Hawkes DL. The mesophilic and thermophilic anaerobic digestion of coffee waste containing coffee grounds. *Water Res.* 1996;30:371-377.
18. Kiyohara Y, Miyahara T, Mizuni O, Noike T, Ono K. A comparative study of thermophilic and mesophilic sludge digestion. *Water Environ Manage.* 2000;14:150-154.
19. Moen G, Stensel HD, Lepistö R, Ferguson J. Effect of solids retention time on the performance of thermophilic and mesophilic digestion. *Water Environ Res.* 2003;75:539-548.

Manuscript received Feb. 13, 2004, and revision received May 3, 2005.